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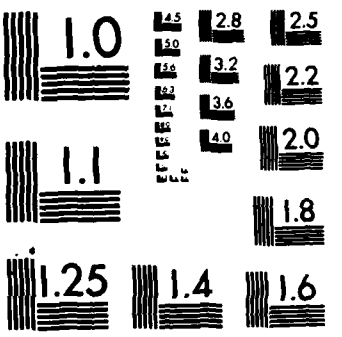

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Infrared Photothermal Radiometry (IPTR) is a sensitive technique for non-contact spectroscopy and inspection. A modulated beam of photons (or other particles) produces temperature transients in a sample; the corresponding transients in the infrared thermal radiation emitted from the sample are analyzed. This can provide absolute absorption coefficients, as well as information on thermal diffusivity, layered structure, and dimensions. Variations of IPTR sre possible with detection. These variations are reviewed. The recent technique of pulsed IPTR with remote

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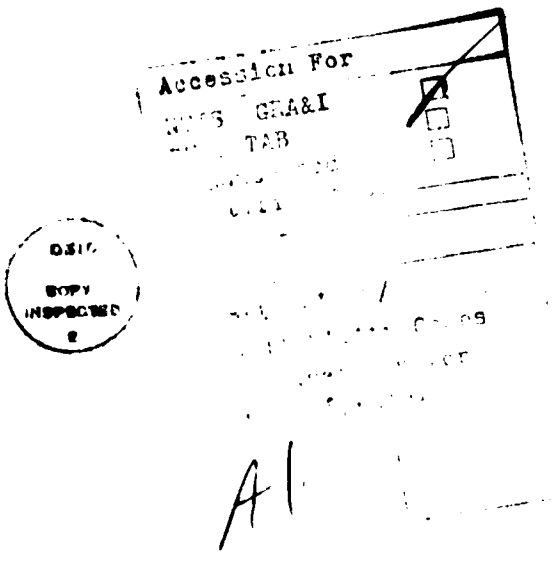

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Infrared Photothermal Radiometry

by

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Physics

Research Report

INFRARED PHOTOTHERMAL RADIOMETRY

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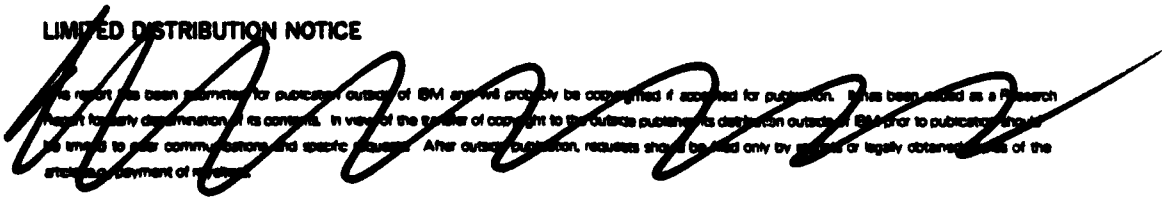

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RJ 4253 (46691) 4/10/84
Physics

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ABSTRACT: Infrared Photothermal Radiometry (IPTR) is a sensitive technique for noncontact spectroscopy and inspection. A modulated beam of photons (or other particles) produces temperature transients in a sample; the corresponding transients in the infrared thermal radiation emitted from the sample are analyzed. This can provide absolute absorption coefficients, as well as information on thermal diffusivity, layered structure, and dimensions. Variations of IPTR are possible with continuously-modulated or pulsed excitation, and with transmission or back-scattering detection. These variations are reviewed. The recent technique of pulsed IPTR with back-scattering detection is described in more detail, and some important single-ended remote sensing applications are discussed.

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1. INTRODUCTION

The photo-thermal (PT) effect is the conversion of electro-magnetic energy, in part or in full, into heat energy in a material. PT effect can be detected directly, *e.g.*, by "calorimetry,"[1,2] or indirectly, *e.g.*, by the opto-acoustic effect,[3] monitoring refractive index variations causing defocusing[4,5] or deflection of probe beams,[6-10] surface distortions,[11,12] spectroscopy,[13] and radiometry. This paper is concerned with the last technique, *i.e.*, infrared photo-thermal radiometry (IPTR). IPTR relies on the detection of variations in the infrared thermal radiation emitted from a sample that is excited by electromagnetic radiation (typically from a laser or from an arc lamp) of varying intensity or wavelength. The advantages of IPTR compared to other PT monitoring techniques are the following: (1) It is totally noncontact, so that measurements can be made on "difficult" samples, like those in vacuum, high pressure, high temperature, or other hostile environments. (2) With the use of IR detectors of fast rise time, thermal or mechanical properties of thin film samples can be measured in much shorter times (or even in "real time") compared to conventional methods. (3) Absolute values for absorption coefficients, thermal diffusivity, or thickness of samples can be obtained in a noncontact manner. (4) IPTR performed in the back-scattered mode appears to be useful for single-ended remote-sensing of samples that may be ~km away, *i.e.*, back-scattering IPTR may provide a new PT LIDAR (Light Detection And Ranging).

There are four variations of IPTR techniques that have been reported in the literature as indicated in Table I. These variations can be classified according to the excitation mode (continuously modulated or pulsed) and to the detection mode (transmission or back-scattered). In this paper, we first review the various modes of IPTR. Then, we examine in more detail the new pulsed back-scattered IPTR, the mathematical basis of this technique, as well as its applications.

TABLE I
Examples of Various Modes of Infrared Photo-thermal Radiometry

Source	Detection	
	Transmission	Back-scattering
Continuous modulated	Busse[15](1980) Busse and Eyerer[17](1983)	Nordal and Kanstad[21](1979) Nordal and Kanstad[23](1981)
Pulsed	Deem and Wood[18](1962) Taylor[20](1972)	Tam and Sullivan[25](1983) Leung and Tam[26](1984)

2. VARIATIONS OF IPTR

The technique of IPTR involves a method of modulated heating of the sample and a method of detecting the IR thermal radiation emitted somewhere from the sample. The modulation of the excitation beam (which can be photon, electron, microwave, *etc.*) is usually either a continuous modulation with about 50% duty cycle, or a pulsed modulated with very low duty cycle, but high peak power. The observation spot can in principle be anywhere on the sample; however, the mathematics is simplest for observation of IR emission emitted backwards from the excitation spot (called back-scattering IPTR here), or emitted from a spot that is "end-on" through the sample thickness with respect to the excitation spot (called transmission IPTR here). Indeed, most workers have used either the back-scattering geometry or the transmission geometry, although Luukkala *et al.*,[14] have demonstrated IPTR with the observation spot being laterally displaced from the excitation spot. Back-scattering IPTR (but not transmission IPTR) can be used for thick or bulky materials or for samples with inaccessible back surface.

The principle of all variations of IPTR is the following. The modulated excitation beam produces a modulated temperature $\theta(\vec{x},t)$ in the sample, where $\vec{x}$ is position and t is time. This modulated temperature profile is dependent on the absorption coefficient α of the excitation beam. Thermal diffusion causes a spatial spreading of the temperature profile, depending on the thermal diffusivity D, and on the boundary conditions. In particular, for thin film samples of thickness L, the thermal diffusion in the thickness direction is hindered when the thermal diffusion length is comparable or larger than L. The modulated IR radiation emitted from the observation region depends on the temperature at that region as well as the mean absorption coefficient α' of the IR light averaged over the detection bandwidth. The above qualitative discussions indicate that the IPTR signal can provide information on the parameters α, D, L, or α' of the sample.

2.1 Continuously Modulated IPTR in Transmission. Transmission IPTR using an argon ion laser of 1W power modulated at 15-30 Hz has been demonstrated by Busse.[15] This technique was first proposed by Cowan[16] in 1961 for thermal diffusivity measurements at high temperatures. In Busse's work, a wedge-shaped piece of aluminum of thickness ranging from 1 to 5 mm is irradiated by the focused laser beam on one surface; the modulated thermal IR emission that is emitted "end-on" from the other surface is detected by a Golay cell. Busse[15] observed that the IR radiometry signal S(L,t) is given by

$$S(L,t) = A \exp(-L/\mu) \exp[i(\omega t - L/\mu)] \qquad (1)$$

where L is the aluminum plate thickness at the observation position, A is an amplitude, and μ is the thermal diffusion length for the laser modulation frequency f, with $\omega=2\pi f$:

$$\mu = [D/\pi f]^{1/2} . \tag{2}$$

Busse's[15] experiment indicates that if S(L,t) is measured for a range of modulation frequency f or for a range of thickness L, the thermal diffusivity of the samples can be derived. Further, if a plate with internal voids is scanned across the laser beam, the transmission IPTR signal changes whenever the transmitted thermal wave crosses a void. This provides a means of nondestructive subsurface imaging of defects, and Busse found that the monitoring of the phase angle of the IPTR signal is more reliable for imaging compared to the monitoring of the amplitude of the IPTR signal. This is because the signal amplitude also depends on the absorption or reflection properties of the sample surface (through the factor A in Eq. (1)), while the phase angle only depend on the thermal diffusivity along the transmission path. Busse and Eyerer[17] have later applied the technique for remote, nondestructive characterization of structures and processes in polymers.

2.2 Pulsed IPTR in Transmission. The first IPTR experiment was performed by Deem and Wood[18] in the pulsed mode in transmission. This method was based on the earlier flash thermometry technique developed by Parker *et al.*,[19] who excited the front surface of a metal plate by a flash lamp, and detected the temperature transient at the back surface by a thermocouple. Deem and Wood[18] replaced the contact thermocouple by a noncontact PbS IR detector, and the flash lamp by a 5 Joule ruby laser. They have used their "laser flash thermal diffusivity apparatus" to measure thermal diffusivity D of nuclear reactor fuel materials at high temperatures. For measurements of such types of "hostile" samples, noncontact techniques like IPTR is of course preferred to avoid corrosion and aging problems. The value of D (which is related to the quality of the composite fuel material) can be obtained by analyzing the shape of the transmission IPTR signal, as already shown in the flash thermometry measurement of Parker *et al.*[19] The simplest data analysis method is to measure the "half-time" ($t_{1/2}$) required for the transmitted IPTR signal to reach half of the maximum. In this case, D can be shown[19] to be

$$D = 1.38\ L^2/\pi^2\ t_{1/2} \tag{3}$$

where L is the sample thickness.

Deem and Wood's[18] technique, called pulsed IPTR in transmission here, is also frequently simply called the "flash diffusivity" or "flash radiometry" technique in the literature. Several authors have taken advantage of this noncontact technique to measure D at high temperatures. For example, Taylor[20] has used it to measure D of resin-bonded carbon from 20°C to 1650°C, and observe annealing of the material at 850°C. He also could make measurements under various vacuum or high pressure conditions.

The "shape analysis" in the flash diffusivity technique corresponds to the "phase angle" analysis of Busse.[15] In the flash excitation, the excitation beam is modulated by a broad spectrum of Fourier modulation frequencies. In all cases of subsurface imaging, the authors preferred to analyze the shape or the phase of the IPTR signal rather than the amplitude, because the latter can be affected by surface conditions of the sample.

2.3 Continuously Modulated IPTR in Back-scattering. Nordal and Kanstad[21-23] first demonstrated continuously modulated IPTR in back-scattering. In their earlier work,[21] they used a continuous CO_2 laser (of power ~50 mW) modulated at 77 Hz at 50% duty cycle as the excitation source, and an InSb photoconductor as the IR radiometry detector. The laser can be tuned from 9.2 to 10.8 μm. They showed that absorption spectra for powdered samples at high temperatures can be obtained by scanning the CO_2 laser and plotting the IPTR signal amplitude as a function of laser wavelength. This is quite valuable, since absorption spectroscopy of an opaque and/or highly light-scattering specimen is traditionally very difficult, especially if the specimen is at high temperature. In subsequent work, Nordal and Kanstad[23] has shown that a conventional high pressure Xe lamp/monochromator combination is sufficient for IPTR spectroscopy. The excitation power level at the sample is less than 1 mW. A PbSnTe IR detector is used for observing the thermal radiation from the excitation region. By scanning the excitation monochromator, they obtained optical absorption spectra for a broad variety of untreated and strongly light-scattering samples, including Nd_2O_3 powder, blood, and a green leaf. Luukkala[24] has shown that the technique of Nordal and Kanstad[21-23] is not only good for spectroscopic detection, but also for subsurface imaging applications as well.

2.4 Pulsed IPTR in Back-scattering. We have recently developed[25-27] the new technique of pulsed IPTR in back-scattering. The advantages of back-scattering detection have already been noted earlier. The advantage of pulsed excitation is that with the use of a pulsed laser beam, sufficient PT heating can be produced in a distant sample to provide a detectable IR radiometry signal, so that remote sensing of distant samples is possible. Also, with pulsed excitation at low duty cycle (say $\lesssim 10^{-6}$), a negligible amount of steady-state sample heating is produced, and hence signal interpretation is simplified. Furthermore, the pulse excitation permits the use of "gating" techniques for suppression of noise during the excitation pulse. This is because during the excitation pulse, instrumental scattering and electromagnetic interference effects are possible; however, the IPTR signal usually has a much broader width compared to the excitation pulse width, and a "detection gate" can be turned on after the excitation pulse. Such advantages of pulsed excitation compared to continuously modulated excitation are already known in other techniques; for example, Tam and Coufal[28] have discussed such comparisons in connection with opto-acoustic spectroscopy.

The method of pulsed IPTR is indicated in Fig. 1. We have used various pulsed excitation sources, for example a short duration (full width at half maximum=8 nsec) N_2 laser with 1 mJ output energy at 337 nm. Most of our experiments are concerned with the new technique of back-scattered IPTR as shown for beam path (a) in Fig. 1, although some of our work is also performed with transmission IPTR as indicated in beam path (b) in Fig. 1. The thermal IR radiation from the sample is refocused onto a HgCdTe detector with a rise time of 0.5 μsec, and the IPTR signal in back-scattering ($S_B(t)$) or in transmission ($S_T(t)$) is accumulated on a transient recorder (Tektronix 7854).

We[25] have applied this pulsed IPTR in back-scattering for novel absolute absorption spectroscopy of opaque solids and liquids. This is possible because the magnitude of the absorption coefficient α of the excitation beam is related to the "steepness" of the initial temperature profile produced in the sample. Large α corresponds to a steeper temperature profile near the sample surface, causing faster cooling of the surface and hence faster decay of the back-scattered IPTR signal.

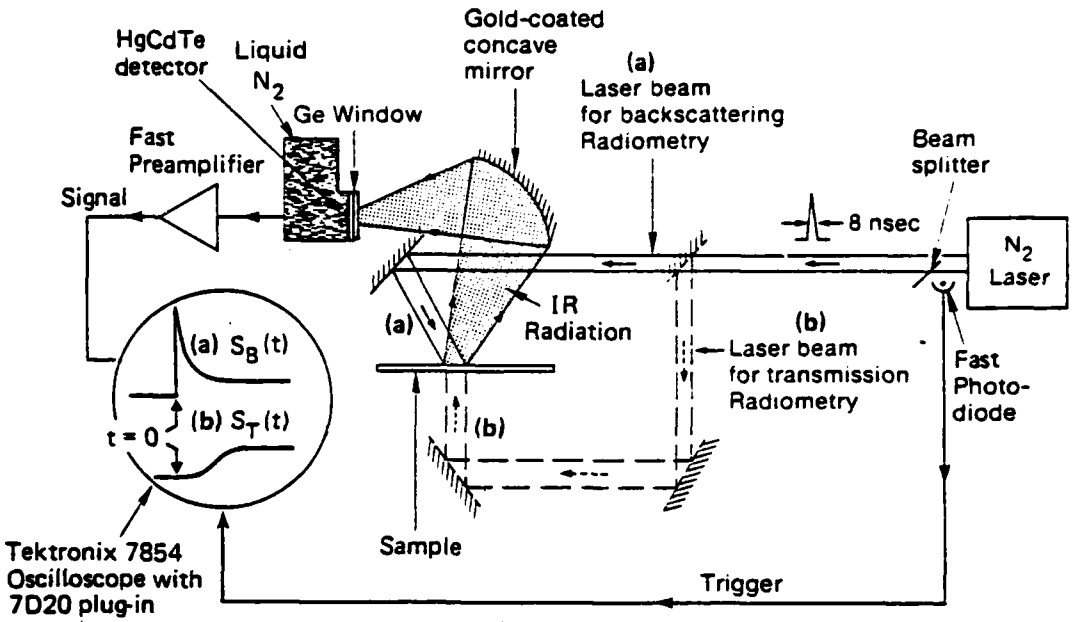

Fig. 1. Experimental setup for both single-ended back-scattering (laser beam represented by solid lines) and double-ended transmission (laser beam represented by dash lines) flash radiometry measurements.

By analyzing the shape of the IPTR signal from bulk samples, Tam and Sullivan[25] showed that α can be obtained under some conditions if the thermal diffusivity D is known.

Another very important application[25] is the remote sensing of the layered structure of a sample by pulsed IPTR. To show this, we coat a black rubber substrate with a 45 μm thick polyester film that is transparent in the wavelength of the excitation beam. For this layered structure, the pulsed IPTR exhibits two components: a prompt component that decays with the same rate as the bare substrate but with decreased magnitude, and a delayed component that peaks up at a time t_d after the firing of the laser and decays afterwards. We further found that t_d is related to the thickness ℓ and the thermal diffusivity D_ℓ of the coating by

$$t_d = \ell^2/4D_\ell \, . \tag{4}$$

These observations indicate that the prompt component is due to IR thermal radiation emitted from the irradiated substrate, and the delayed component is due to IR thermal radiation emitted from the top coating surface due to heat diffusion from the illuminated substrate surface.

We have also[25] demonstrated that pulsed IPTR is useful to sense the degree of powder aggregation which affects the "effective" thermal conductivity. The pulsed laser is used to irradiate black carbon-loaded epoxy powders of different degree of compactness. We find that the pulsed IPTR signal for the loose powder stops decaying after some time indicating that inter-particle heat transport is very slow. However, the pulsed IPTR signal for a compact powder exhibits a continuous decay as in a neat solid.

We have later[26] studied the pulsed IPTR for thin films. The signal decays differently in two different time regimes, as indicated in Fig. 2. For a uniform homogeneous thin film of thickness L, we can define the characteristic thermal diffusion time τ_L (as Parker *et al.*[19]):

$$\tau_L = L^2/\pi^2 D \, . \tag{5}$$

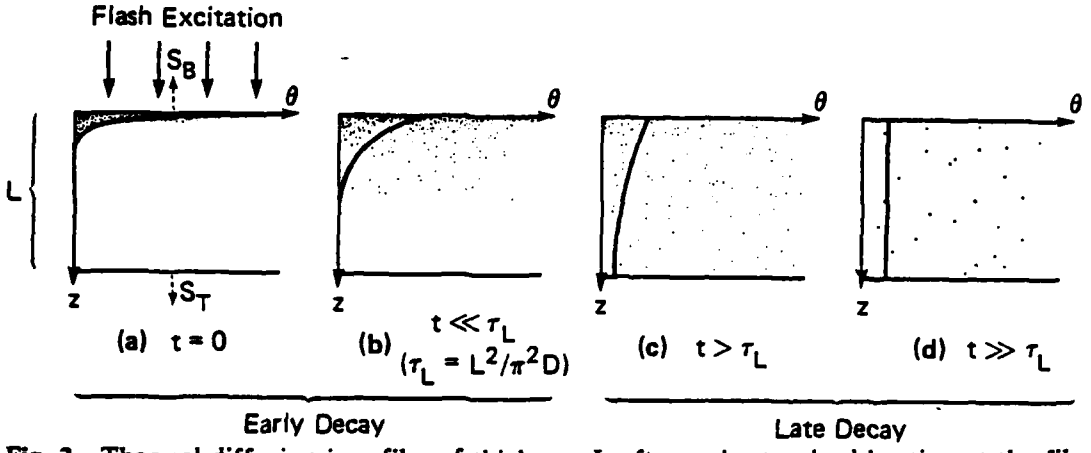

Fig. 2. Thermal diffusion in a film of thickness L after a short pulsed heating at the film surface. The local temperature increase above ambient (θ) is plotted at several times (t), and also indicated by the density of the small black dots.

For short times, when $t<<\tau_L$ (see Fig. 2), the presence of the back surface is "not felt", and heat diffusion proceeds as a semi-infinite solid. In this case, the rate of the surface temperature decay depends only on the absorption coefficient α of the excitation light and D; hence the early decay of the IPTR signal depends only on α, α', and D, where α' is the absorption coefficient of the IR radiation averaged over the detection wavelength range (here, we assume αL, $\alpha' L$ are both $>>1$). For late times, when $t>>\tau_L$ (see Fig. 2), the presence of the back surface plays a major role in the cooling rate of the front surface, and the decay of the IPTR signal for $t>>\tau_L$ depends only on L and D, as first shown by Parker *et al.*[19] for sufficiently opaque plates. The above qualitative discussion can be mathematically quantified,[27] and some numerical results are shown in Figs. 3 and 4. Figure 3 shows the theoretical shapes of the pulsed IPTR signal $S_B(t)$ for two samples of identical material (stainless steel) but different thickness: L=0.005 cm (dashed line) and L=∞. We see that the two signals totally overlap at early times; however, after τ_L=63.3 μsec for the L=0.005 cm sample, the two signals start to deviate, with the thin-film signal stop decaying while the "thick plate" signal continue to decay. These effects have been verified experimentally.[26,27] Figure 4 shows how the theoretical shapes of $S_B(t)$ depend on the absorption coefficients α and α', with fixed thickness and thermal diffusivity. We see that at early times (*i.e.*, $t<<\tau_L$), the stronger absorption coefficients cause a steeper decay of the IPTR signal. However, at late times ($t>>\tau_L$) the decay curves for different values of α and α' are overlapping, showing that the late decay rate is only dependent on L and D, in agreement with the analysis of Parker *et al.*[19] To sum up, the back-scattered IPTR signal $S_B(t)$ is sensitively dependent on α, α' and D for $t<<\tau_L$, and sensitively depend on L and D for $t>>\tau_L$. Theoretical fitting[27] of the observed $S_B(t)$ for a sample can provide values of α, α', D, and L. Such measurements have the distinct advantages of being single-ended, remote-sensing, and nondestructive.

Spectroscopy of the sample is possible with pulsed back-scattered IPTR by scanning the wavelength of the excitation laser and monitoring the magnitude of the back-scattered IPTR signal. An example of such absorption spectrum is shown in Fig. 5.

3. THEORY OF PULSED IPTR

Detailed theory of the continuously modulated IPTR technique has been given by several authors, for example, Santos and Miranda[29] and Tom *et al.*,[30]

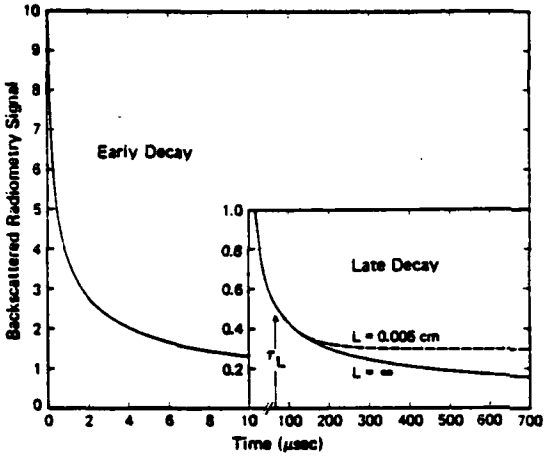

Fig. 3. Theoretical profile of the single ended back-scattered flash radiometry signal showing the effect of sample thickness L. Dash line is for L=0.005 cm and solid line is for L=∞. In both curves, α, α' and D are taken as 2×10^4 cm^{-1}, 1×10^4 cm^{-1} and 0.04 cm^2/s, respectively, which are typical values for stainless steel. Note the scale changes for the late decay in the inset.

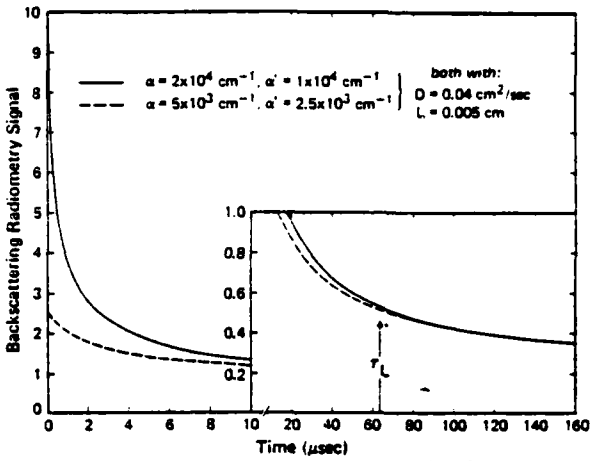

Fig. 4. Theoretical profile of the back-scattered radiometry signal showing the effect of absorption coefficient α and α'. Dash lines is for $\alpha=5\times10^3$ cm^{-1} and $\alpha'=2.5\times10^3$ cm^{-1}; solid line is for $\alpha=2\times10^4$ cm^{-1} and $\alpha'=1\times10^4$ cm^{-1}. In both curves, L and D are taken as 0.005 cm and 0.04 cm^2/s, respectively. Note scale changes for the late decay in the inset.

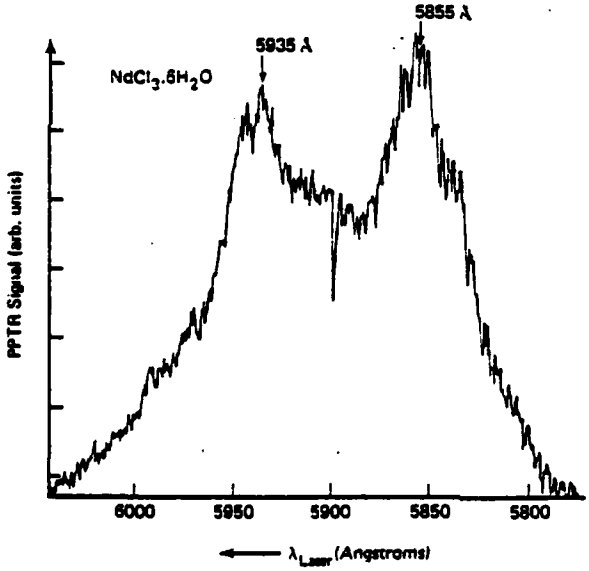

Fig. 5. Pulsed IPTR spectrum with back-scattering detection for $NdCl_3.6H_2O$. This is obtained by scanning the excitation pulsed dye laser over the Rhodamine 6G tuning range (5770Å to 6030Å). The numbers marked are in Angstrom units.

and their results will not be described here. We have[27] recently derived the theoretical pulsed IPTR signal in back-scattering ($S_B(t)$) and in transmission ($S_T(t)$) for a short excitation laser pulse and fast IR detector response time.

$$\begin{bmatrix} S_B(t) \\ S_T(t) \end{bmatrix} = \frac{AK}{L}\left\{(1-e^{-\alpha L})(1-e^{-\alpha' L})\right.$$

$$\left. \pm 2\begin{bmatrix} 1 \\ e^{-\alpha' L} \end{bmatrix} \sum_{n=1}^{\infty} \left(\frac{1-(-1)^n e^{-\alpha L}}{1+\frac{n^2\pi^2}{\alpha^2 L^2}}\right)\left(\frac{1-(-1)^n e^{\mp\alpha' L}}{1+\frac{n^2\pi^2}{\alpha'^2 L^2}}\right) e^{-n^2 t/\tau_L} \right\} \quad (6)$$

where the equation for the upper/lower qualities in the two square brackets correspond to the upper/lower ± signs. Here, A is a constant depending on the flash pulse energy and the thermal properties of the sample, and K is a constant depending on the emissivity and steady temperature of the sample. Equation (6) is basically the quantitative expression for the qualitative statements in Section 2.4. Equation (6) can be shown[27] to approach simple limits when L is large. In this case, $S_T(t) \longrightarrow 0$ as $L \longrightarrow \infty$, and

$$S_B(t) \underset{L\to\infty}{\longrightarrow} \frac{AK\alpha\alpha'}{\alpha'^2-\alpha^2}\left\{\alpha' e^{t/4\tau_\alpha}\left(1-\mathrm{erf}\sqrt{t/4\tau_\alpha}\right)\right.$$

$$\left.-\alpha\, e^{t/4\tau_\alpha}\left(1-\mathrm{erf}\sqrt{t/4\tau_{\alpha'}}\right)\right\} \qquad (7)$$

where $\tau_\alpha = (4\alpha^2 D)^{-1}$, $\tau_{\alpha'} = (4\alpha'^2 D)^{-1}$ and erf is the error function.[31] Equation (7) is symmetrical with respect to the absorption coefficients α and α'. It further simplifies if one of the absorption coefficient is much larger than another, *e.g.*, $\alpha' >> \alpha$. In this case:

$$S_B(t) \underset{\substack{L\to\infty \\ \alpha'>>\alpha}}{\longrightarrow} AKe^{t/4\tau_\alpha}\left(1-\mathrm{erf}\sqrt{t/4\tau_\alpha}\right). \qquad (8)$$

Equation (8) is precisely the form we have used in our earlier[25] pulsed IPTR for obtaining absolute values of α at the excitation wavelength for "semi-infinite" samples. In Eq. (6), we have neglected the effects due to the finite excitation pulse width and the finite IR detector rise time. Such effects have been incorporated in the work of Leung and Tam,[27] where experimental data to support our theoretical $S_B(t)$ and $S_T(t)$ profiles are also presented.

4. CONCLUSIONS

Techniques of IPTR rely on the detection of modulated infrared thermal radiation from a sample that is excited by a modulated beam of energy. The two types of detection modes discussed here are back-scattering or transmission, and these are compared in Table II. The two types of modulations commonly used are continuous modulation (~50% duty cycle) or pulsed ($\lesssim 10^{-6}$ duty cycle) modulation. The pulsed mode can be regarded as the frequency-multiplexed version of the continuously modulated mode and they are compared in Table III. These variations of IPTR are reviewed. The recent technique of pulsed IPTR in back-scattering detection is examined in more detail; this is the only variation that is suitable for remote sensing, and the various new applications and the theoretical basis are discussed. IPTR techniques also have obvious industrial and medical inspection applications, to the extent that a "Laser Inspect System" consisting of a modulated continuous NdYAG laser and an InSb IR detector is now commercially available.[32]

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TABLE II
Comparison of "Back-scattering" and "Transmission" Detection for Radiometry

Item	Back-scattered Radiometry	Transmissive Radiometry
Applicability for films with backside being nonassessible	Yes	No
Time-dependent signal shape	Monotonic decreasing if film is homogeneous; if layered, may have delayed peaks.	Delayed peak and subsequent decay.
Depth-profiling capability	Yes, because smaller depth is sensed by early signal.	Not easy, because the signal is mainly sensitive to an average thermal diffusivity.
Suitability for remote-sensing	Yes, because all measurement equipments are on one side of the sample (*i.e.*, single-ended).	Not easy, because measurement equipments are needed on both sides of the sample (*i.e.*, double-ended).

TABLE III
Comparison of Continuous Modulated and Pulsed Excitation for Radiometry

	Continuous Modulated	Pulsed
Feature	High duty cycle, low peak power	High peak power, low duty cycle
Steady background heating	Usually substantial	Usually small
Suitability for remote sensing of samples (*e.g.*, 1 -Km away)	No	Yes (using pulsed lasers)
Tight focusing of the excitation laser beam	Usually required	Usually not required
Time-gating of detection signal for"noise" reduction	Not possible	Possible
Important parameter to be measured	Phase angle of signal with respect to the excitation (hence, only 1 data point)	Entire shape of the radiometry transient signal (hence, many data points)

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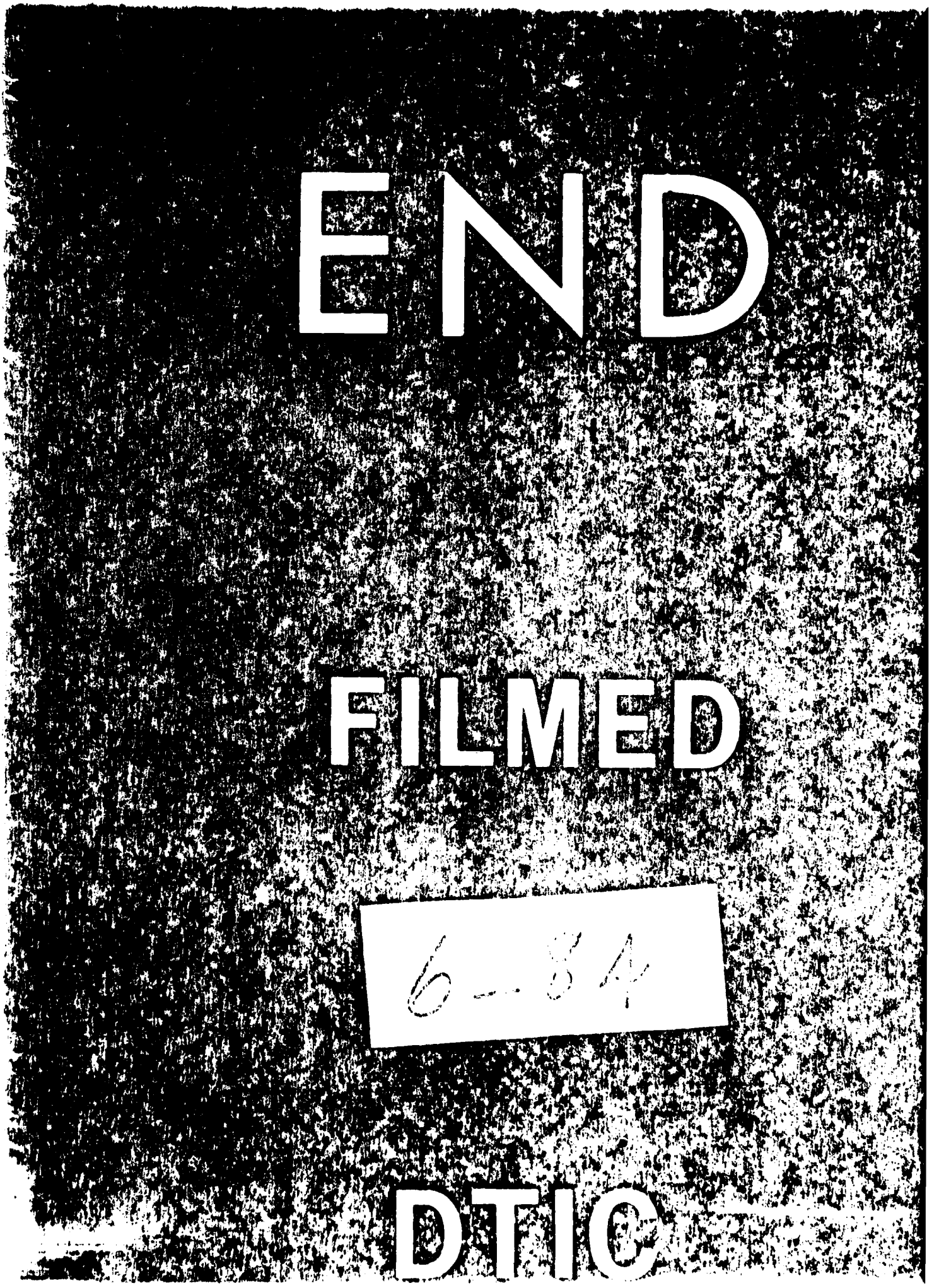
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